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IS 11557 (1986): 2- Naphthylamine - 3 : 6 : 8 -
Trisulphonic Acid, Technical [PCD 9: Organic Chemicals
Alcohols and Allied Products and Dye Intermediates]



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Indian Standard

SPECIFICATION FOR
2 - NAPHTHYLAMINE - 3 : 6 : 8 - TRISULPHONIC
ACID, TECHNICAL

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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Indian Standard

SPECIFICATION FOR 2 - NAPHTHYLAMINE - 3 : 6 : 8 - TRISULPHONIC ACID, TECHNICAL

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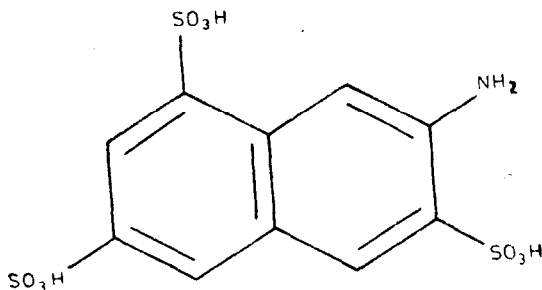
Indian Standard

SPECIFICATION FOR 2 - NAPHTHYLAMINE - 3 : 6 : 8 - TRISULPHONIC ACID, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 29 January 1986, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 2-Naphthylamine - 3 : 6 : 8 - trisulphonic acid ($C_{10}H_6O_3NS_3$) is used as an intermediate for reactive dyes. It is represented by the following structural formula:



2-Naphthylamine - 3 : 6 : 8 - trisulphonic acid
(Molecular Mass 383)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 2-naphthylamine - 3 : 6 : 8 - trisulphonic acid technical.

*Rules for rounding off numerical values (revised).

2. REQUIREMENTS

2.1 Description — 2-Naphthylamine - 3 : 6 : 8 - trisulphonic acid, technical shall be pale yellow to yellow moist material soluble in dilute alkaline solution.

2.2 The material when tested according to the methods given in Appendix A shall also comply with the requirements given in Table 1. Reference to the relevant clauses of Appendix A is given in col 4 of Table 1.

TABLE 1 REQUIRMENTS FOR 2-NAPHTHYLAMINE - 3 : 6 : 8 - TRISULPHONIC ACID, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST REF TO CLAUSE No. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Matter insoluble in dilute sodium carbonate solution, percent by mass, <i>Max</i> (on dry basis)	0.2	A-1
ii)	Assay (on dry basis), percent by mass, <i>Min</i>	75	A-2
iii)	Amido G. acid, percent by mass, <i>Max</i>	0.5	A-3

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (see IS : 2552-1970*) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

- Name of the material;
- Name of the manufacturer and his recognized trade-mark, if any;
- Tare, net and gross mass; and
- Batch number.

3.2.1 The containers may also be marked with the ISI Certification Mark.

*Specification for steel drums (galvanized and ungalvanized (*first revision*)).

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standards conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and the quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Preparation of Test Samples — The method of preparing representative sample of the material shall be as prescribed in 3 of IS: 5299-1969*.

4.2 Number of Tests

4.2.1 Each individual sample shall be tested for assay.

4.2.2 Test for the remaining characteristics as prescribed in 2 and Table 1 shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 For Individual Samples — The lot shall be declared as conforming to the requirement of assay, if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 For Composite Samples — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (see 4.2.2), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

5. TEST METHODS

5.1 Tests shall be carried out according to the method prescribed in Appendix A, as indicated in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Method of sampling and test for dye intermediates.

†Specification for water for general laboratory use (second revision).

APPENDIX A

(Clause 2.2 and Table 1)

METHODS OF TEST FOR 2-NAPHTHYLAMINE - 3 : 6 : 8 - TRISULPHONIC ACID, TECHNICAL

A-0. PREPARED SAMPLE

A-0.1 Dry the material at $105 \pm 1^\circ\text{C}$ to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests.

A-1. DETERMINATION OF MATTER INSOLUBLE IN DILUTE SODIUM CARBONATE SOLUTION

A-1.1 Reagent

A-1.1.1 Sodium Carbonate Solution — Dissolve 10 g of sodium carbonate in water contained in 100-ml measuring flask and make up to the mark.

A-1.2 Procedure — Weigh accurately 20 g of the sample and transfer it to a 500-ml beaker. Add 300 ml of hot water followed by 10 percent sodium carbonate solution (A-1.1.1) to make the solution alkaline to brilliant yellow paper. Heat the solution until the material is completely dissolved and filter it through sintered glass crucible (G_3). Wash the residue with hot water till free from alkali, dry at 100°C to constant mass, cool and weigh. Preserve the filtrate and washings for assay (see A-2.1).

A-1.3 Calculation

$$\frac{\text{Matter insoluble in sodium carbonate}}{\text{solution, percent by mass}} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the sample taken for the test.

A-2. ASSAY

A-2.1 Procedure — Cool the filtrate and washings (see A-1.2) to room temperature. Mix well and dilute with water to 500 ml in a volumetric flask. Pipette out 50 ml aliquot in a one litre beaker. Add 400 ml

ice-cold water followed by 35 ml of concentrated hydrochloric acid. Titrate with $\frac{N}{10}$ sodium nitrite solution at 15°C, while stirring, using potassium starch iodide papers, to a 10 minute end point.

A-2.2 Calculation

$$\text{Assay, percent by mass} = \frac{V \times N \times 383}{M}$$

where

M = mass in g of the sample taken for test;

V = volume in ml of sodium nitrate solution used for titration; and

N = normality of sodium nitrate solution used for titration.

A-3. DETERMINATION OF AMIDO G. ACID

A-3.0 Outline of the Method — Amido G. Acid is determined by using descending paper chromatographic technique.

A-3.1 Apparatus

A-3.1.1 Developing Chamber

A-3.1.2 Micropipette

A-3.1.3 UV lamp

A-3.2 Reagents

A-3.2.1 Ammonium Hydroxide Solution — 1 percent (m/v).

A-3.2.2 Developer — n -propanol water + benzene (65 : 35 : 5) ($v/v/v$).

A-3.2.3 Amido G. Acid — pure.

A-3.2.4 2-Naphthylamine - 3 : 6 : 8 - trisulphonic acid — Free from Amido G. Acid.

A-3.3 Procedure — First, prepare standard solutions of 2-naphthylamine 3:6:8-trisulphonic acid containing known amounts of Amido G. Acid by accurately weighing 1.0 g of the material (A-3.2.4) into each of three 100-ml volumetric flasks and adding 3.0 ml, 4.0 ml and 5.0 ml of 0.10 percent solution of Amido G. Acid (A-3.2.3) in 1.0 percent ammonium hydroxide solution to flask No. 1, 2 and 3 respectively. Dissolve the contents of the flask in ammonium hydroxide solution (A-3.2.1). Thus there shall be 3 solutions of 0.3, 0.4 and 0.5 percent Amido G. Acid content. In the fourth flask weigh about 1.0 g of the prepared sample under test (see A-0.1), dissolve in ammonium hydroxide solution and dilute to 100 ml with ammonium hydroxide solution.

A-3.3.1 Place 5 microlitre spot of the four solutions using micro-pipette in the same line to a distance of about 4 cm on filter paper (Whatman No. 1 or equivalent). Place the paper in a descending paper chromatographic glass jar containing the developing reagent and previously saturated with the same developer. Allow the solvent to run in a descending manner at room temperature for about 40 cm from the spot. This will take about 15 hours. Take out the paper after 40 cm run and dry the solvent completely.

The chromatogram so developed is observed under ultra violet light when the separated spots characteristic of constituents may be identified by their colour as under:

<i>Content</i>	<i>Zone</i>	<i>Rf Value</i>	<i>Colour</i>
Unknown	I	0.05	Red
Unknown	II	0.25	Yellowish
2-Naphthylamine - 3 : 6 : 8 - trisulpho- nic acid,	III	0.34	Bluer
Amido G. Acid	IV	0.50	Blue

A-3.4 Report — Report Amido G. Acid content as that which is nearest in intensity to the standard. In case the colour intensity does not come in the range of standard spots, repeat the whole procedure using different percentage of Amido G. Acid.